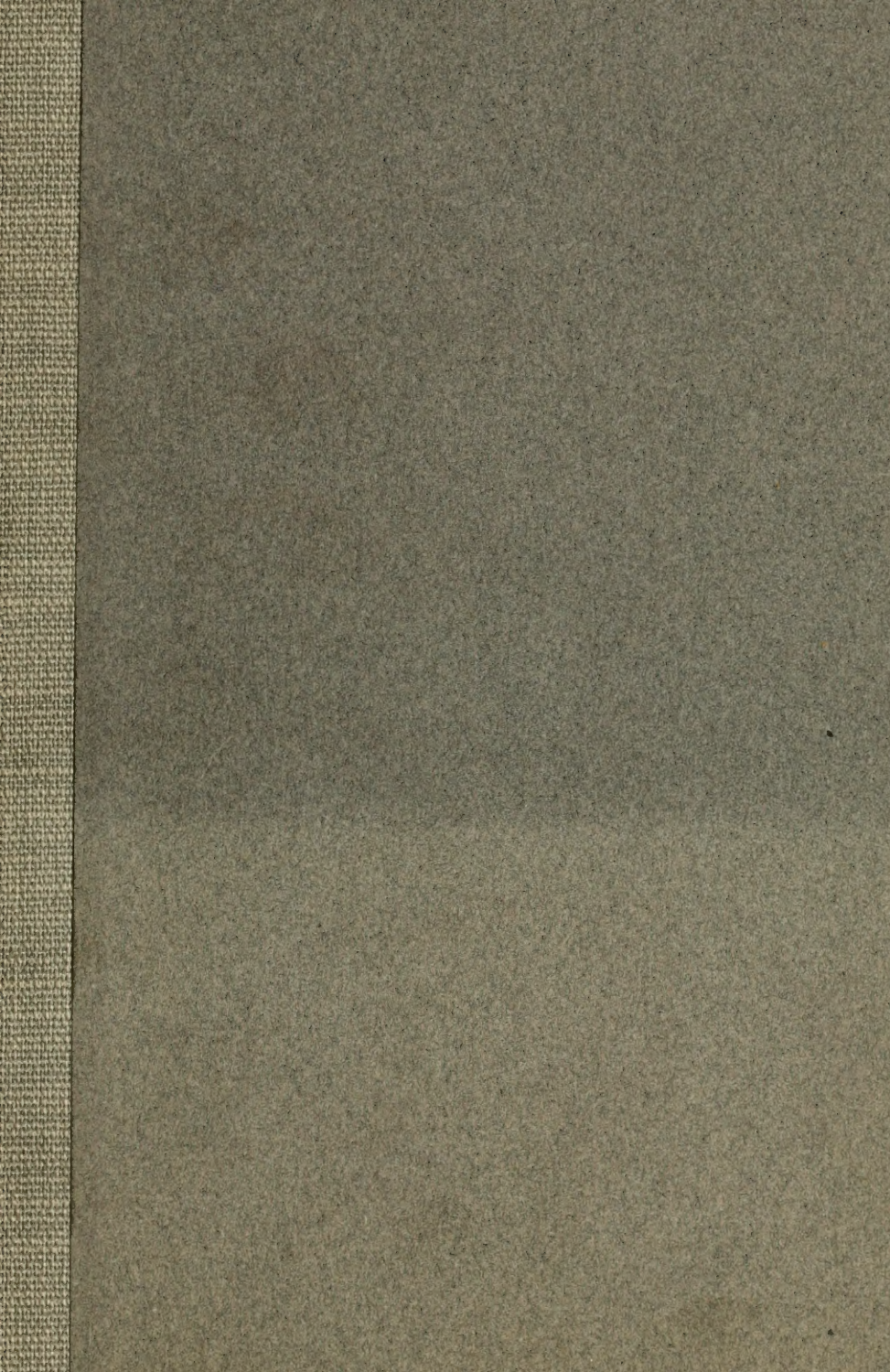
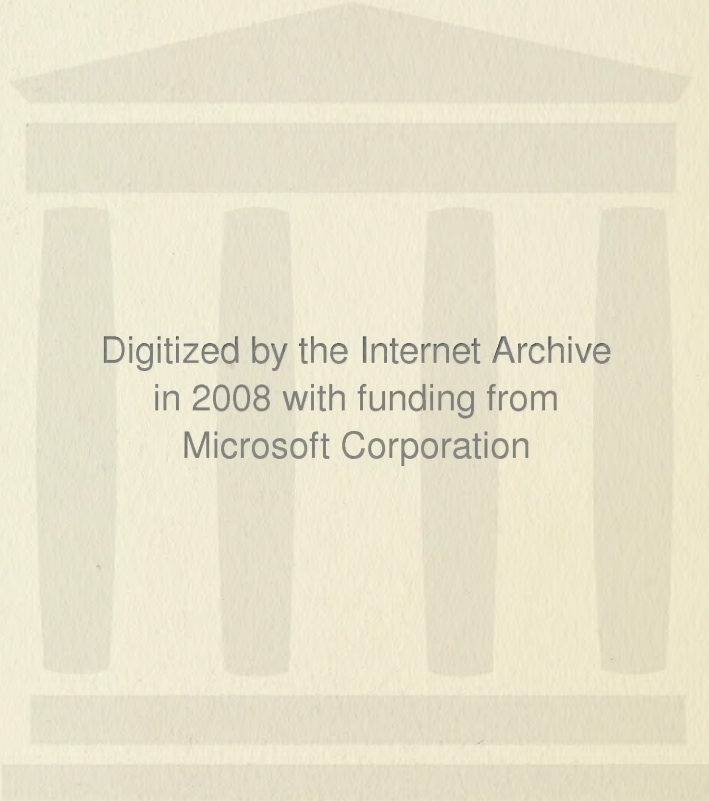






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R. T. STULL, Acting Director

DEFORMATION TEMPERATURES OF SOME PORCELAIN GLAZES

BY

R. T. STULL AND W. L. HOWAT

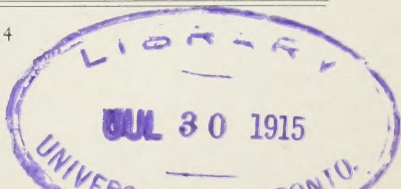
A TYPE OF CRYSTALLINE GLAZE AT CONE 3

BY

C. C. RAND AND H. G. SCHURECHT

PUBLISHED BY THE UNIVERSITY OF ILLINOIS, URBANA

1913-1914



DEFORMATION TEMPERATURES OF SOME PORCELAIN GLAZES

R. T. STULL AND W. L. HOWAT, URBANA, ILL.

The group of glazes studied comprises ten horizontal series designated by letters from A to J, each series consisting of ten members. The group of one hundred members covers the following limits represented by the four corner glazes:

TABLE I—FORMULA OF CORNER GLAZES

GLAZE	K ₂ O	CaO	Al ₂ O ₃	SiO ₂
A-1	0.3	0.7	0.40	2.0
A-10	0.3	0.7	0.40	6.5
J-1	0.3	0.7	0.85	2.0
J-10	0.3	0.7	0.85	6.5

TABLE II—BATCH WEIGHTS

	BRANDY- WINE FELDSPAR	WHITING	ENG. NO. 20 BALL CLAY	N. C. KAOLIN	FLINT	Al ₂ (OH) ₃
A-1	167.4	70.0	12.9	12.9		
A-10	167.4	70.0	12.9	12.9	270.0	
J-1	167.4	70.0	12.9	12.9		70.2
J-10	167.4	70.0	70.9	70.9	216.0	

Different members in the group were made by molecular blending of the four extremes. These were applied to bisque wall tile, set in saggars in a down draft kiln and burned to cone 9 in 40 hours.

Cones were also made from the glazes and their deformation temperatures determined in a platinum resistance furnace, the temperatures being measured by a platinum, platinum-rhodium thermocouple and a Leeds-Northrup direct reading potentiometer, (accurate to 3°C).

The time-temperature curve followed in all determinations is shown in Figure 1. The temperature was raised to 1200°C. in 120 minutes. Beyond this the temperature rise was 2½ degrees per minute. A number of deformation tests made on duplicate Seger cones gave the following results: cone 4—1212°C., cone 6—1255°C., cone 8—1290°C.

Deformation-temperature readings were made on two or more cones of each glaze. The variation was rarely over 5°C., and in the majority of tests, duplicate cones gave the same temperature readings.

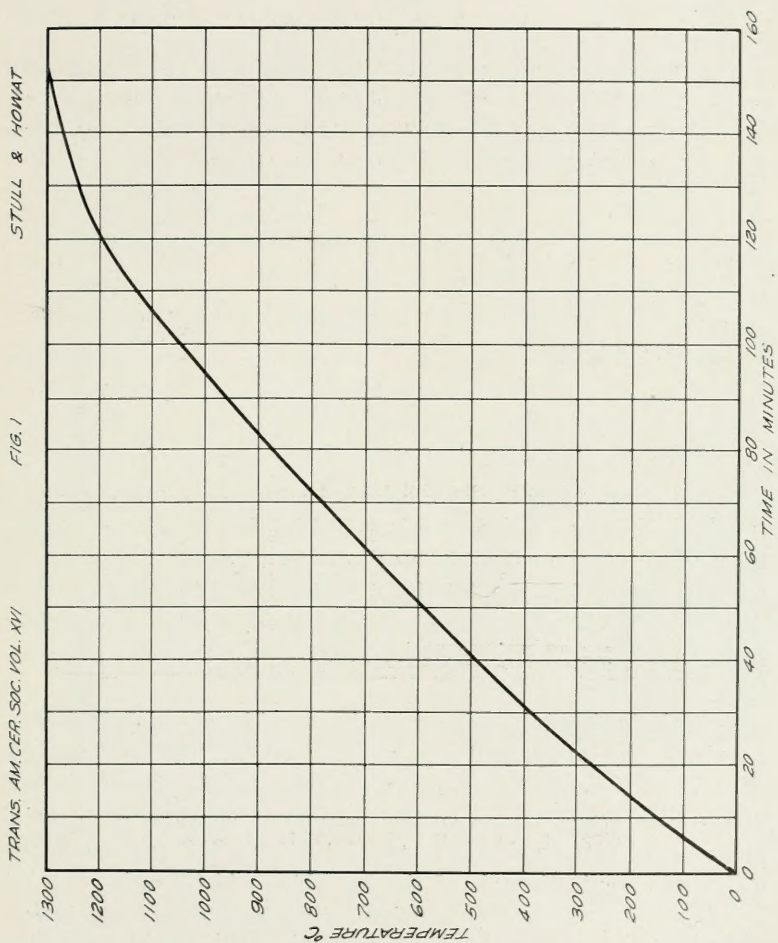
TABLE III—DEFORMATION TEMPERATURES COVERING THE LIMITS

0.3 K₂O : 0.40 to 0.85 Al₂O₃ : 2.0 to 6.5 SiO₂
0.7 CaO :

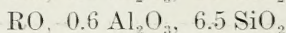
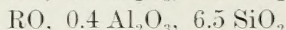
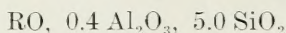
	1	2	3	4	5	6	7	8	9	10	MOLECULES Al ₂ O ₃
J	1277	1246	1232	1235	1247	1252	1248	1260	1267	1265	0.85
I	1275	1240	1228	1230	1240	1235	1245	1247	1250	1252	0.80
H	1272	1245	1232	1230	1230	1232	1235	1235	1245	1245	0.75
G	1272	1240	1228	1228	1232	1232	1233	1237	1235	1247	0.70
F	1267	1238	1225	1225	1225	1225	1228	1235	1235	1245	0.65
E	1232	1225	1225	1222	1220	1225	1228	1235	1245	1245	0.60
D	1230	1225	1225	1227	1230	1230	1240	1245	1248	1252	0.55
C	1232	1228	1228	1228	1228	1230	1240	1248	1252	1255	0.50
B	1235	1230	1228	1233	1235	1245	1254	1252	1257	1270	0.45
A	1232	1232	1240	1245	1245	1255	1255	1268	1272	1277	0.40
MOLECULES SiO ₂	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	6.5	

The average temperature readings for two or more cones of each glaze of the group are given in Table III. The results of the burn and the iso-deformation lines are represented graphically in Figure 2, the deformation-temperature being indicated in degrees centigrade on each line.

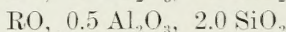
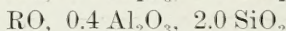
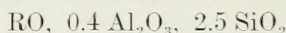
The RO is constant for all glazes. The molecular variations of SiO₂ are plotted along the abscissa and the molecular variations of Al₂O₃ on the ordinate.



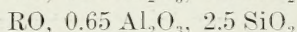
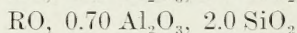
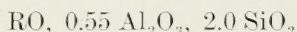
In the lower right corner are the devitrified glazes between the limits:



In the lower portion of the devitrified area the glazes were crazed. In the center of the field are the bright glazes which were considered matured. Bright glazes which were crazed are found in the lower left corner within the limits:



At the left of the field a small group of matured mats are found between limits:



In the upper part of the field the glazes were under fired.

The difference between max and min deformation temperatures is 57°C ., the softest one deforming at 1220°C . having the formula, $\text{RO, } 0.6 \text{ Al}_2\text{O}_3, 4.0 \text{ SiO}_2$. The member at the upper left corner ($\text{RO, } 0.85 \text{ Al}_2\text{O}_3, 2.0 \text{ SiO}_2$) and the one at the lower right corner ($\text{RO, } 0.4 \text{ Al}_2\text{O}_3, 6.5 \text{ SiO}_2$) deformed at the max temperature 1277°C .

Each horizontal series may be considered as being composed of the components, glaze and SiO_2 . The broken line CD passes through the deformation-eutectic of each of the ten glaze— SiO_2 series. In a vertical direction, consider each series made up of glaze and Al_2O_3 , the dotted line EF represents the deformation-eutectic axis of the ten glaze— Al_2O_3 series.

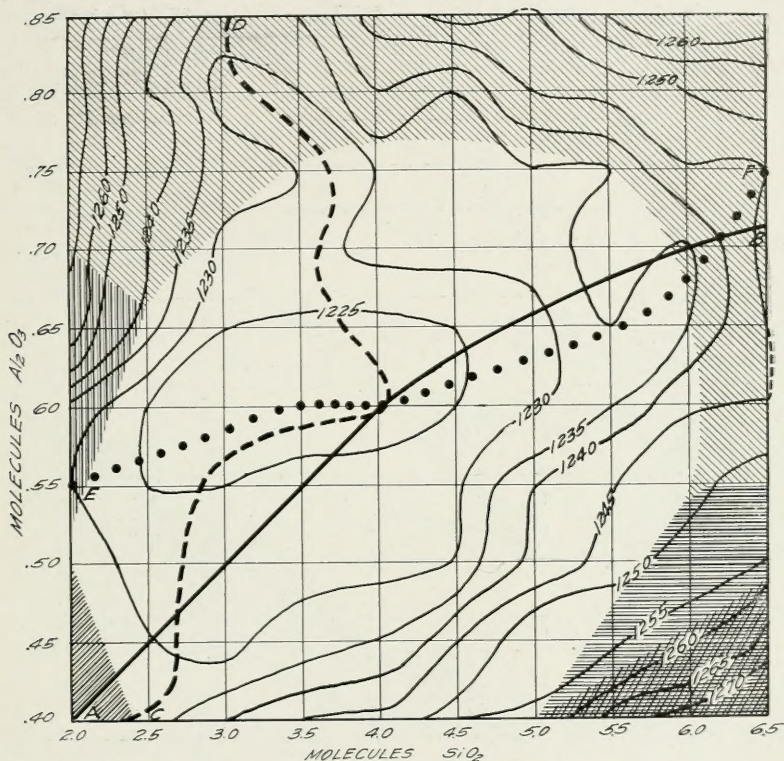
These two axes (CD and EF) cross at the point of lowest deformation temperature (group eutectic). Its deformation temperature is ten degrees higher than the indicated temperature of Seger cone 4. The glazes whose formulae correspond to cones 4, 5 and 6 deformed at 1228°C , 1240°C and 1245°C respectively.

The line AB is the high gloss axis plotted according to the appearance of the glazed trials. The gloss axis follows roughly parallel to the glaze-SiO₂ deformation-eutectic axis up to the group eutectic. Beyond this point it deflects and follows along

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FIG. 2

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CONE NINE BURN

 CONSTANT {
 .3K₂O
 .7CaO

BRIGHT

MATT

IMMATURE

DEVITRIFIED

CRAZED

DEVITRIFIED CRAZED

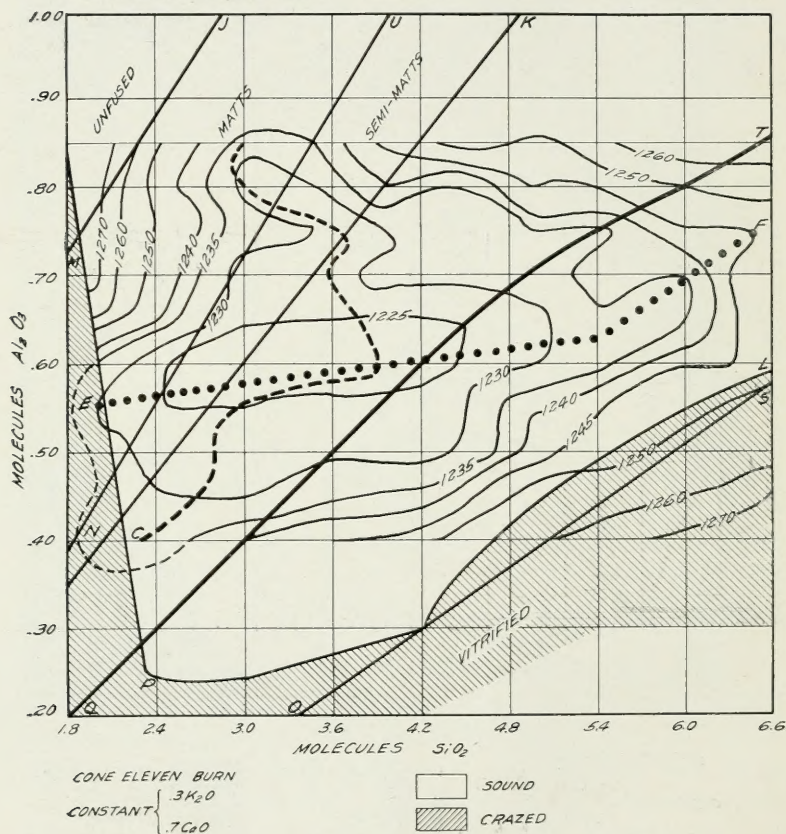
the glaze-Al₂O₃ deformation-eutectic axis. The group deformation-eutectic lies near the center of the field of best glazes, and the quality of the glazes decreases in all direction away from this eutectic point. The general formulae of the best glazes as shown by the trials are:

RO-0.60 Al_2O_3	4.0 SiO_2	Deformation temp. = 1220°C.
RO-0.55 Al_2O_3	3.5 SiO_2	Deformation temp. = 1227°C.
RO-0.55 Al_2O_3	4.0 SiO_2	Deformation temp. = 1230°C.
RO-0.60 Al_2O_3	3.5 SiO_2	Deformation temp. = 1222°C.

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FIG. 3

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The difference between the deformation-temperatures of these glazes and the temperature to which they were fired (cone 9) is 80°C to 90°C, or a difference of 4 to 4½ cones. For the purpose of comparison the iso-deformation temperature lines are plotted on the field of porcelain glazes burned at cone 11 and

previously reported,¹ Figure 3. The high gloss axis QT lies to the right of the glaze-SiO₂ eutectic axis and crosses the glaze-Al₂O₃ eutectic axis close to the eutectic member of the group. The best glazes in this group are found in close proximity to the group eutectic, the same as in the cone 9 burn. Not only does the group eutectic lie near the center of the area of best glazes, but it is also located at a safe distance away from devitrification, crazing, matness and immaturity.

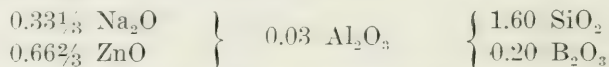
Ceramic Laboratories,
University of Illinois.

¹ Influences of variable Silica and Alumina on Porcelain Glazes of Constant RO. *Trans. Amer. Cer. Soc.*, Vol. xiv, pp. 62-70.

A TYPE OF CRYSTALLINE GLAZE AT CONE 3

C. C. RAND AND H. G. SCHURECHT, URBANA, ILL.

The glazes under consideration are of a type designed to mature about cone 3 to 4. The Al_2O_3 is maintained constant throughout at .05 equivalent and is introduced as Pikes No. 20 English ball clay. In general the group resembles Worcester's¹ best raw clay glaze. His formula was



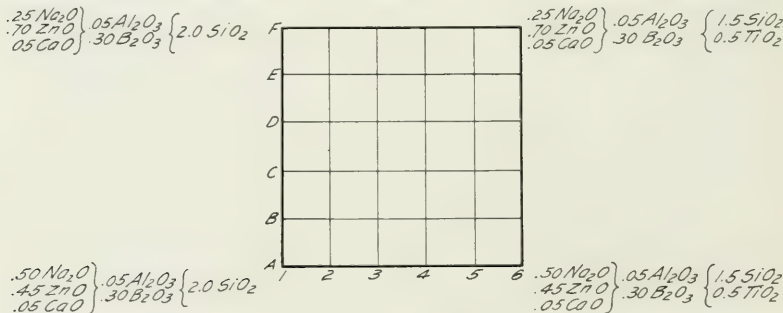
He concludes, however, that .05 Al_2O_3 generally seems the most favorable and that many German formulæ call for this amount.

A group of 36 glazes was made with a view to determine the effect of varying ZnO against Na_2O along the ordinate, and rutile against flint along the abscissa.

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FIG. 1

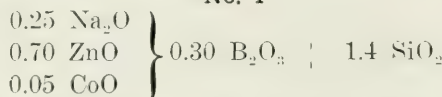
RAND & SCHURECHT



The arrangement of the group with the formulæ of the four corners are shown in Fig. 1, the vertical series being designated by numbers, the horizontal series by letters.

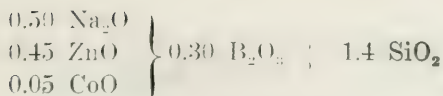
Two frits of the following compositions were used:

No. 1



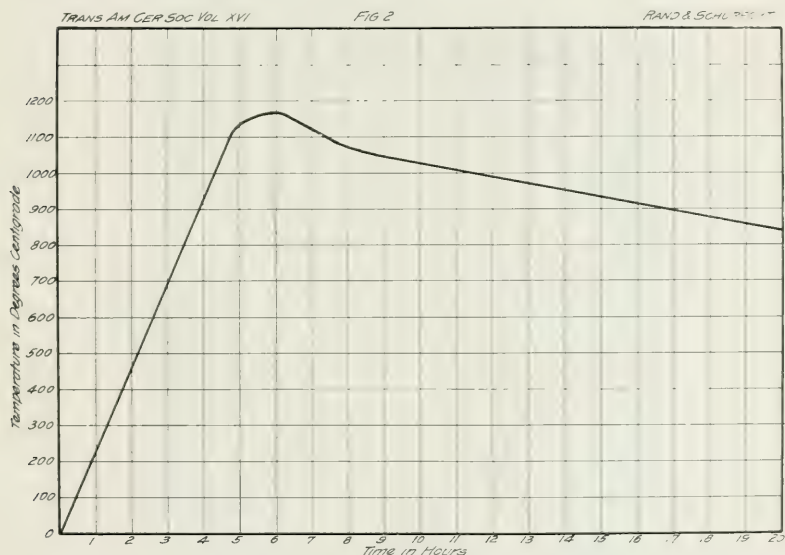
¹ Vol. V *Trans. Amer. Cer. Soc.*, p. 150. Function of Alumina in Crystalline Glaze.

No. 2



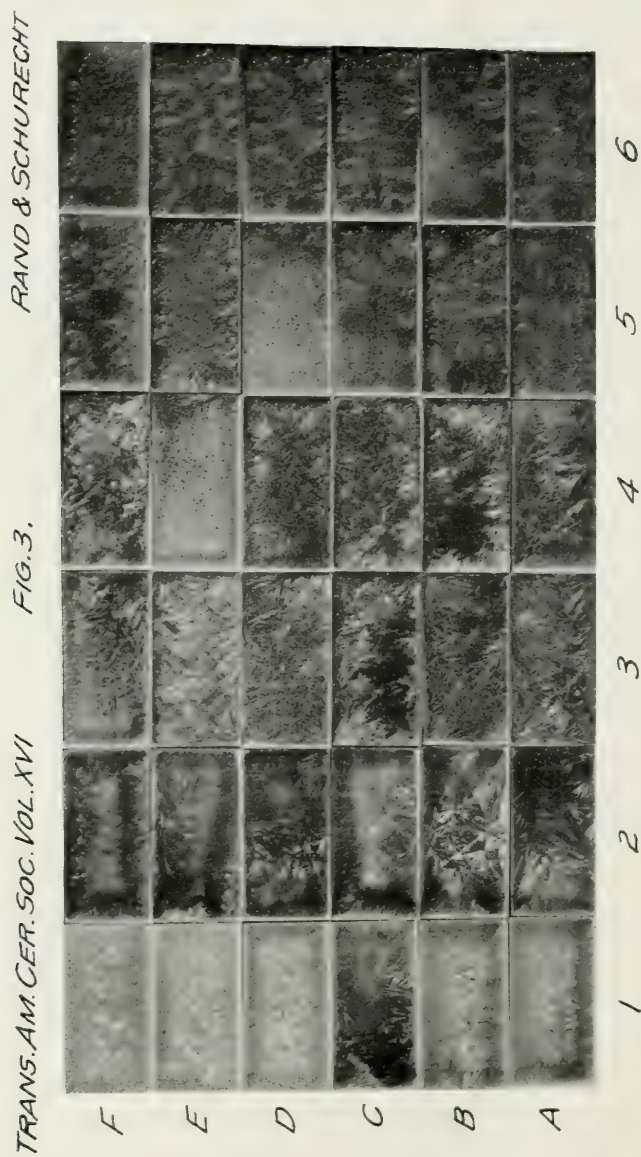
These were each intimately mixed and ground in small ball mills, fused, quenched in water, dried and ground to pass 80 mesh sieve.

The four corner glazes were ground wet until they passed 120 mesh sieve, and the remaining glazes blended from them in molecular proportions.



The glazes were applied to two sets of biscuit tile by dipping, and burned to cone 3 in 5 hours in a round, down-draft, open-fired oil kiln. The fires were put out when the finishing temperature was reached and the kiln allowed to cool with the damper closed.

The results were not satisfactory, as only a few crystalline patches appeared. These patches increased noticeably as the content of ZnO increased. High ZnO also appeared to give a deeper blue as would be expected. E 3 showed the most crystallization.



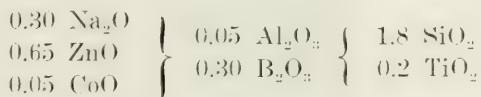
The failure to obtain good results was attributed to too rapid cooling, too thin coating of the glaze, and perhaps slight under burning.

Next two sets of trials were dipped, care being taken to obtain a thick coating of the glaze, and burned in the same kiln to cone 4, following the heating and cooling curve shown in Figure 2. The pyrometer showed a temperature of 1170°C, when cone 4 went down.

From a crystallization standpoint, the results, Fig. 3, were highly satisfactory. Every glaze showed a large number of crystals and in many cases was a solid mass of crystals of varying sizes. The variation of ZnO and Na₂O seems to have little, if any, effect upon either crystallization or color.

Increase in TiO₂ has a marked effect upon both. As TiO₂ increased the crystals became smaller, and more numerous, most of the high rutile glazes consisting of a mass of small interlocking crystals. At 0.0 TiO₂ and at 0.1 TiO₂, a good blue color is shown, but from 0.1 TiO₂ up, the blue is partially and in some cases almost totally absent. Bronze patches are quite prominent, due possibly to iron impurities in the rutile.

The two sets of trials are very nearly identical. Glaze E 3, with the formula

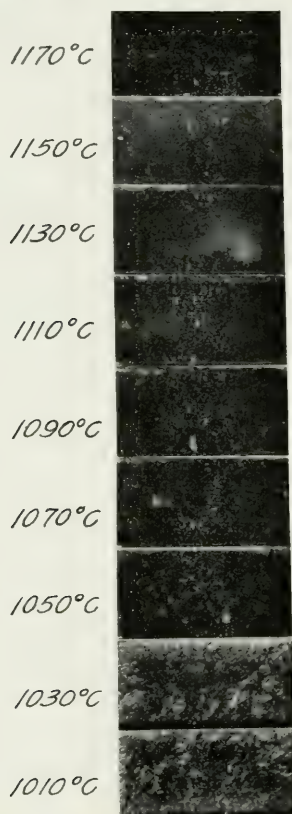


again appears to contain the most crystals and for this reason was selected as the glaze to use in making draw trials with a view to noting different stages of crystallization.

The glazes were dipped and burned in the same manner as before, a number of trials of E 3 being placed where they could be drawn. One was drawn at the finishing point, and one every 20° as the cooling progressed. The third trial drawn showed crystals around the edges. The amount of crystallization increased steadily for four trials. The next trial at 1030°C showed a very great increase, the glaze consisting of a mass of crystals. It is possible that this is due to the crystallization of an eutectic.

That is the compound forming the crystals shown first continued crystallizing out until the melt reached the composition of the eutectic mixture, when the whole mass crystallized. (Fig. 4.)

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One set of trials was placed on edge in this last burn. They failed to show as much crystallization as those lying down, as the glaze had run off to a great extent. However, good results were obtained on two small vases to which the glaze had been applied in a very thick coat, though here also, much of the glaze had been lost.

